

International Comparison in the Pressure Range 20–100 MPa

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Abstract

An international comparison in the pressure range 20–100 MPa has been carried out under the auspices of the high-pressure working group of the Comité Consultatif pour la Masse et les grandeurs apparentées (CCM) of the Comité International des Poids et Mesures (CIPM). The Standards Laboratories of 13 countries have participated in this comparison, which took place during the period 1981–1985. This paper presents a résumé of the comparison.

The transfer standard used was an oil-operated pressure balance. Each laboratory determined the effective area of the piston-cylinder assembly of this balance in the pressure range 20–100 MPa. The results of the measurement of the effective area extrapolated to zero applied pressure agreed within 204 parts per million (ppm) for all 13 laboratories (9 laboratories agreed within 53 ppm). The results of the measurement of the effective area at 100 MPa agreed within 414 ppm for all 13 laboratories (8 laboratories agreed within 78 ppm).

1. Introduction

During a meeting held in June 1980 at the Bureau International des Poids et Mesures (BIPM), the high-pressure Working Group of the Comité Consultatif pour la Masse et les grandeurs apparentées (CCM) agreed to organize an international comparison in the pressure range 20–100 MPa. When the comparison began, 13 laboratories confirmed their wish to participate. Because of the large number of participants, a comparison following a "petal" scheme was organized in three phases. Each phase entailed evaluation of the transfer standard by the Pilot Laboratory, the Laboratoire National d'Essais (LNE), measurement of the transfer standard by each of four

participating laboratories, then re-evaluation of the transfer standard by the Pilot Laboratory at the end of the phase. Detailed results of each of the three phases of the comparison have been published as BIPM reports [1–3]; the present article is a résumé thereof.

The timetable and the metrologist principally responsible for the work at each laboratory were as follows:

Laboratoire National d'Essais (LNE), Paris, France (May 1981); J. C. Legras

Istituto di Metrologia "G. Colonnetti" (IMGC), Turin, Italy; G. F. Molinar

Physikalisch-Technische Bundesanstalt (PTB), Braunschweig, Federal Republic of Germany; J. Jäger

National Physical Laboratory (NPL), Teddington, United Kingdom; S. L. Lewis

National Bureau of Standards (NBS), Gaithersburg, USA; V. E. Bean

LNE (August 1982)

Bundesamt für Eich- und Vermessungswesen (BEV), Vienna, Austria; R. Lewisch

Ceskoslovenský Metrologický Ústav (CSMU), Bratislava, Czechoslovakia; A. Kepř

Aeronautical Research Institute (FFA), Bromma, Sweden; L. Rydström

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LNE (April 1984)

National Research Laboratory of Metrology (NRLM), Ibaraki, Japan; S. Yamamoto

National Institute of Metrology (NIM), Beijing, China; Sheng Yi-Tang

Amt für Standardisierung, Meßwesen und Warenprüfung (ASMW), Berlin, GDR; K. Möbius

Gostandard-VNIIFTRI, Moscow, USSR; V. M. Borovkov

LNE (August 1985)

G. F. Molinar, the chairman of the Working Group, was responsible for co-ordinating the intercomparison.

The comparison was carried out on a blind basis, so the results are given here in a comparative form to facilitate further phases with additional participants in the future.

For the measurement of pressure in the range above atmospheric, the primary standard in general use is the pressure balance (or piston gauge), where the pressure is derived from the application of a known gravitational force balanced against an upward force generated by the action of the system pressure on a known area. This area is provided by a carefully matched piston-cylinder assembly, and is termed the effective area of the assembly. The determination of the effective area, especially its dependence upon pressure due to the elastic distortion of the piston and cylinder, forms the major source of uncertainty in establishing high-pressure standards. Dissemination of pressure measurements in this pressure range is also achieved using pressure balance. The natural choice for a transfer standard for this intercomparison was therefore a pressure balance, the measured parameter being the effective area of its piston-cylinder assembly as a function of pressure.

2. Details of the Transfer Standard

The transfer standard was a Desgranges et Huot, type 5300 S, oil-operated pressure balance which had been placed at the Working Group's disposal by the manufacturer. The piston-cylinder assembly, of nominal effective area 5 mm^2 , consisted of a tungsten carbide cylinder and a steel piston. The stainless-steel weights, of total mass 50 kg, enabled pressure of up to 100 MPa to be measured.

In use the piston was rotated by a motorized eccentric-drive, and the temperature of the piston-cylinder assembly was monitored using a platinum resistance thermometer. The thermal expansion coefficient of the assembly was determined in two ways: LNE measured directly the changes in effective area of the piston-cylinder assembly whilst housed in a climatic chamber, and NPL measured the linear thermal expansion coefficients of two samples of the materials of the piston and cylinder; the results from two methods were in very close agreement, namely $14.7 \times 10^{-6}/^\circ\text{C}$ and $14.65 \times 10^{-6}/^\circ\text{C}$, respectively, for the change in effective area. To minimize possible variations in the transfer standard's performance due to change in the properties of the pressure medium, samples of the same oil (diethylhexylsebacate) were sent to each participant.

A detailed procedures document, prepared by the present authors, was circulated with the transfer standard to ensure that all participants followed the same working method; also provided by the Pilot Labora-

tory were calibration data for the weights, the platinum resistance thermometer and the thermal expansion coefficient to be used for the piston-cylinder assembly.

A second, identical, piston-cylinder assembly was calibrated by the Pilot Laboratory and held in reserve in case of accident to the one circulated. It was not necessary to use the reserve assembly for the comparison itself, but it provided useful additional data on the effective-area stability of this particular type of assembly.

3. Participants' Standards

There are two important areas in which the participants' standards differ, namely the material of construction of the piston and cylinder, and the design of the cylinder. Both features influence the distortion of the two components when subjected to applied pressure. Table 1 lists these characteristics for each participant. Detailed discussions of the three main designs may be found elsewhere [4, 5], but they are briefly as follows:

simple: the applied pressure acts on only the *inside* of the cylinder;

re-entrant: the applied pressure acts on both the *inside* of the cylinder, and on the *outside* of it over part of its working length;

controlled-clearance: a subsidiary pressure system applies a controlled, variable pressure to the outside of the cylinder, compensating for the distortion effects due to the internal pressure.

Table 1 also lists in very general terms the method of derivation of the distortion coefficients and, where appropriate, references to the publications describing the standards. As stated above, the major area of difficulty in the measurement of effective area is in establishing its dependence upon applied pressure, i.e., the pressure distortion coefficient λ .

4. Measurement and Calculation Methods

Each laboratory carried out a preliminary assessment of the transfer standard to ensure that it met certain criteria, e.g. the piston and cylinder were carefully checked for magnetism, the direction of rotation of the piston was reversed at the lowest pressure to test for rotational dependence, etc.

The participant then determined the effective area of the piston-cylinder assembly at 20°C , using the data provided by the Pilot Laboratory for all other parameters, e.g. mass of the loading weights, calibration of the temperature sensor, thermal expansion coefficient

Table 1. General details of the reference standards used by the participating laboratories

	Material of construction	Design type	Method of determination	Ref.
LNE	WC/WC	Controlled clearance	Flow leak and variation of jacket pressure	[6]
IMGC	WC/WC	Simple	Comparison with low-pressure balances	[7]
PTB	WC/WC	Simple	Comparison with a 50 MPa standard, the λ of which had been calculated	[8]
NPL	Steel/Steel	Simple	Comparison with standards whose λ had been derived from the Similarity Method	[9, 10]
NBS	Steel/Steel	Controlled clearance	Variation of jacket pressure from null clearance	[5]
BEV	Steel/Steel	Re-entrant	Calculated	—
CSMU	Steel/Steel	Simple	Calculated	[11]
FFA	WC/WC	Re-entrant	Manufacturer's data	—
EAM	Steel/WC	Simple	Comparison with another standard	—
NRLM	Steel/WC	Controlled clearance	Variation of jacket pressure from null clearance	[12]
NIM	Steel/Steel	Simple	Calculated	[14]
ASMW	Steel/Steel	Simple	Calculated	[13]
VNIIFTRI	Steel/Steel	Simple, but used with an intensifier	Calculated	—

of the assembly, properties of the oil, etc. The measurements were made in five pressure cycles by direct comparison (cross floating) between the laboratory's standard and the transfer standard. Each pressure cycle consisted of 17 measurements at 9 applied pressures between 20 MPa and 100 MPa, at intervals of 10 MPa. All the effective-area data were sent to the Pilot Laboratory to ensure analysis on a common basis.

The effective area, A_p , at 20°C was calculated from the following relationships. (The notation used is given in Table 2.) The downward vertical force acting on the piston due to the mass of the weights and the surface tension of the oil is

$$F = mg(1 - \rho_a/\rho_m) + \Gamma C \quad (1)$$

Table 2. Notation used for the transfer standard

Parameter	Symbol
Force applied to the piston	F
Mass of applied weights	m
Gravity	g
Air density	ρ_a
Density of weights	ρ_m
Surface tension of the oil	Γ
Piston circumference	C
Effective area at zero applied pressure and 20°C	A_0
Effective area at applied pressure p	A_p
Pressure distortion coefficient	λ
Temperature of the piston-cylinder assembly	t
Measured pressure	p
Thermal expansion coefficient of area of the piston-cylinder assembly	α
Estimated variance of A_0 , λ	$\sigma_{A_0}^2, \sigma_{\lambda}^2$
Estimated uncertainties of A_0 , λ	$\delta A_0, \delta \lambda$
Measured deviation of A_0 , λ	$\Delta A_0, \Delta \lambda$
Time elapsed since the initial measurement at LNE, i.e. LNE 1, in months	τ

and the effective area of the transfer standard at p and 20°C is given by

$$A_p = F/p [1 + \alpha(t - 20)] \quad (2)$$

where p is the applied pressure (as measured by the laboratory's standard).

In general, the dependence of effective area upon applied pressure, for an assembly of simple form, can be expressed as a linear function of pressure, i.e.,

$$A_p = A_0(1 + \lambda p) \quad (3)$$

where λ is termed the pressure distortion coefficient.

Thus, by calculating A_p from (1) and (2), A_0 and λ may be derived from the least-squares-best-fit straight line

$$A_p = a + b p \quad (4)$$

where $A_0 = a$ and $\lambda = b/A_0$.

5. Analysis of Results

5.1 Stability of the General Parameters

No significant changes were seen in the parameters whose values were provided to the participants by the Pilot Laboratory. It was therefore possible to base the calculation of A_p upon the same values throughout.

5.2 Test of the Linearity Assumption

To test the validity of adopting (3) to express the dependence of the effective area of the transfer standard upon applied pressure, the deviations of the observed

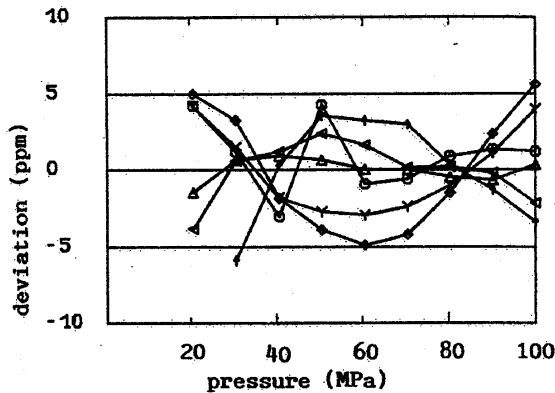


Fig. 1. Deviations in A_p from a least-squares-best-fit straight line, obtained by the first-phase participants, expressed in ppm.
 \triangle LNE 1, \circ IMG, \diamond PTB, ∇ NPL, \uparrow NBS, Δ LNE 2

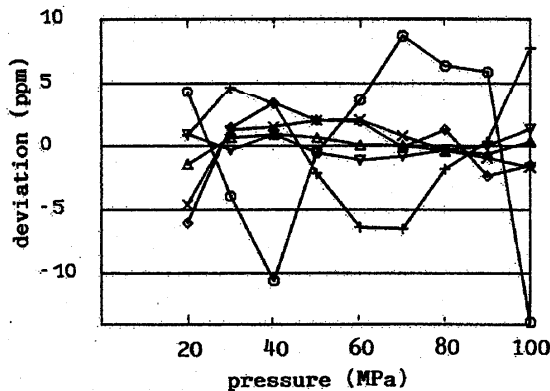


Fig. 2. Deviations in A_p from a least-squares-best-fit straight line, obtained by the second-phase participants, expressed in ppm.
 Δ LNE 2, \circ BEV, \diamond CSMU, $+$ FFA, \times EAM, ∇ LNE 3

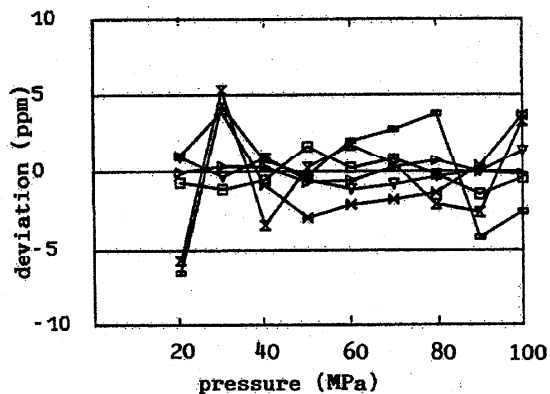


Fig. 3. Deviations in A_p from a least-squares-best-fit straight line, obtained by the third-phase participants, expressed in ppm.
 ∇ LNE 3, \square NRLM, \times NIM, \square ASMW, \times VNIIFTRI, \triangleright LNE 4

values of A_p from the appropriate least-squares-best-fit straight line were derived. This test was initially employed at the end of the first phase, and Fig. 1 shows the deviations, as a function of applied pressure, for each participant; to facilitate comparison, the best-fit straight line of each laboratory has been superimposed on the ordinate axis. The same test was made for the other phases (Figs. 2 and 3).

5.3 Stability of the Effective Area of the Transfer Standard

Analysis of the effective-area data obtained by the Pilot Laboratory for the piston-cylinder assembly of the transfer standard showed significant changes in the effective area, A_0 , with time. The shift, relative to the initial determination, LNE 1, was $+25 \times 10^{-6}$ after 15 months (LNE 2), $+34 \times 10^{-6}$ after 35 months (LNE 3) and $+34 \times 10^{-6}$ after 51 months (LNE 4).

The shift is thought to be due to the fact that the steel pistons were fabricated shortly before the comparison measurements commenced, and the material may not have reached a stabilized condition following its treatment during manufacture. This hypothesis is supported by the fact that similar changes (namely $+35 \times 10^{-6}$, $+50 \times 10^{-6}$ and $+52 \times 10^{-6}$) were seen in the effective area of the second assembly, which was used only for the measurement cycles at LNE, and by the fact that the changes seen decreased appreciably with time. For the purpose of analysing the inter-comparison results, it has therefore been assumed that the changes in effective area with time were of a continuous nature. Small changes were also seen in the values of λ , and the final expression chosen to describe the effective area as a function of both applied pressure and time was:

$$A_p = a_1 + b_1 \log(\tau + c) + dp + e\tau + fp\tau + gp\tau^2. \quad (5)$$

Figure 4 illustrates the deviations in the Pilot Laboratory's observed values from those calculated by the above equation; the deviations can be seen to lie within $\pm 3 \times 10^{-6}$. Equation (5) was therefore used to predict the values of A_0 and λ corresponding to those which the Pilot Laboratory would have obtained at the time the participating laboratory carried out its measurements. These predicted values have been designated

$(A_0)_{PL}$ and $(\lambda)_{PL}$.

5.4 Analysis of the Participants' Data

The data produced by each participating laboratory were forwarded to the Pilot Laboratory, together with an estimate of the laboratory's uncertainty in the measurement of the applied pressures, δp .

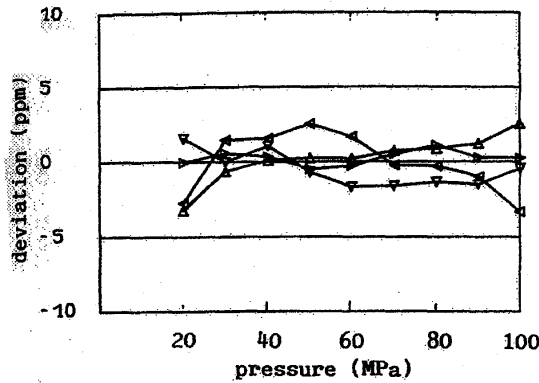


Fig. 4. Deviations in the observed values of A_p , obtained by the Pilot Laboratory, from the values predicted (as a function of time) by Eq. (5), expressed in ppm.
 \circ LNE 1, \triangle LNE 2, ∇ LNE 3, \blacktriangleright LNE 4

In no case did any laboratory's results show any significant systematic differences between the values of A_p obtained on the upward part of the pressure cycle compared with those of the downward part of the cycle. Therefore the analysis of the data has been carried out on the mean value of A_p (generally based on 10 observations) for each applied pressure.

To facilitate comparison between the results obtained from (4), the following differences were computed, thus correcting for the time-dependent changes seen in the transfer standard:

$$(\Delta A_0/A_0)_{\text{LAB}} = [(A_0)_{\text{LAB}} - (A_0)_{\text{PL}}]/(A_0)_{\text{PL}}$$

$$\text{and } \Delta \lambda_{\text{LAB}} = (\lambda_{\text{LAB}} - \lambda_{\text{PL}})$$

where $(A_0)_{\text{LAB}}$ and λ_{LAB} denote the participant's results.

For the final analysis, it was necessary to derive reference values and to present the results in terms of the deviation of each participant's values from these. As there was a wide variation in the uncertainties of measurement claimed by the participants, the reference values were initially calculated from the weighted means of all the results, the weighting being based on the reciprocal of the squares of the uncertainties.

For each participant the uncertainties in their measurement of A_0 and λ were calculated as follows:

$$\frac{\delta A_0}{A_0} = \pm \left[\left(\frac{\delta p}{p} \right)^2 + \left(\frac{\delta m}{m} \right)^2 + \left(\frac{\delta t}{t} \right)^2 \left(\frac{3 \sigma_{A_0}}{A_0} \right)^2 \right]^{1/2} \quad (6)$$

and

$$\delta \lambda = \pm [(3 \sigma_\lambda)^2 + (\delta \lambda')^2]^{1/2} \quad (7)$$

where

$\delta p/p$ is the value of the laboratory's uncertainty in the measurement of p , extrapolated to correspond to zero applied pressure,

δm and δt are the uncertainties with which the Pilot Laboratory's measurements define mass and temperature,

σ_{A_0} and σ_λ are obtained from the least squares best fit of A_p as a linear function of p [equation (4)], and $\delta \lambda'$ is the uncertainty of the pressure distortion coefficient of the laboratory's standard.

Thus, designating each participant's uncertainty in the measurement of A_0 , given by (6), as i , the reference value was calculated from

$$\left[\frac{\Delta A_0}{A_0} \right]_{\text{REF}} = \sum_n \frac{\Delta A_0}{A_0} \frac{1}{i^2} / \sum_n \left(\frac{1}{i^2} \right) \quad (8)$$

where n is the number of participants.

To test the acceptability of each participant's results for inclusion in the weighted mean, (8) was used to calculate a reference value based on $(n-1)$ participants, and the result of the remaining participant was compared with this value; if the difference was less than the uncertainty of that participant, i.e.,

$$\left[\frac{\Delta A_0}{A_0} \right]_{\text{LAB}} - \left[\frac{\Delta A_0}{A_0} \right]_{\text{REF}(n-1)} = k \frac{\delta A_0}{A_0} \quad (9)$$

where $k < 1$, then that participant's value was included in the calculation of a new reference value; those participants whose difference was greater than their uncertainty (i.e., $k > 1$) were not included. This test was used iteratively so that each participant's result (even those which had earlier been omitted) could be re-compared with the new reference value, and re-admitted to the computation if appropriate.

A similar test was used when calculating the reference value for the pressure distortion coefficient, λ .

5.5 Results of the Measurement of the Effective Area at Zero Applied Pressure

Using the above method, a final reference value of $\Delta A_0/A_0$ at 20°C was calculated. Four laboratories' results were excluded from the calculation, as their values did not comply with the acceptability test, their values of k falling in the range $1.26 < k < 3.40$.

The difference between each participant's result and the final reference value is given in Table 3, together with data on their standards; the former is shown graphically in Fig. 5. Overall, the differences of the measurements of the effective area at zero applied pressure are within 204 ppm, and nine results lie within 53 ppm.

5.6 Results of the Measurements of the Pressure Distortion Coefficient

Using the same technique as described above, a reference value was derived for $\Delta \lambda$. Here four laboratories'

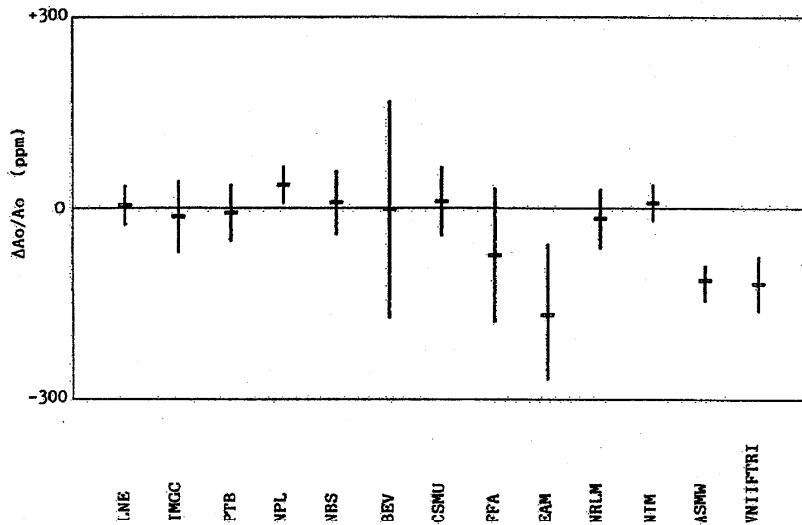


Fig. 5. Deviation of the participants' values of $\Delta A_0/A_0$ from the reference values, expressed in ppm

Table 3. Effective area at zero applied pressure of the participating laboratories' standards, and the deviation (from the reference values) of each participant's measured value of A_0 for the transfer standard

Laboratory	Effective area of the laboratory's own standard at zero applied pressure		Results obtained on the transfer standard	
	(mm ²)	Uncertainty ^a	$\Delta A_0/A_0$ (in ppm)	Uncertainty ^b
LNE	50.2732	27	+ 4.7	31
IMGC	10.02419	40	- 13.3	56
PTB	9.80488	29	- 7.3	44
NPL	30.82795	20	+ 37	29
NBS	32.18869	48	+ 9.4	50
BEV	6.309	159	- 2.0	170
CSMU	10.00043	50	+ 10.8	54
FFA	8.40165	100	- 73.0	105
EAM	4.90204	100	-167	111
NRLM	99.9860	18	- 16.3	46
NIM	10.00451	20	+ 8.7	29
ASMW	20.3325	35	-112	33
VNIIFTRI	20 (nominal)	20	-118	43

^a Uncertainty of measurement expressed in ppm

^b Uncertainty of measurement of the difference (in ppm)

Table 4. Pressure distortion coefficient of the participating laboratories' standards, and the deviation (from the reference value) of each of the participants' measured value of λ for the transfer standard

Laboratory	Pressure distortion coefficient of the laboratory's own standard		Measurement of the pressure distortion coefficient of the transfer standard	
	(MPa ⁻¹) $\times 10^{-6}$	Uncertainty on the value (MPa ⁻¹) $\times 10^{-6}$	$\Delta \lambda$ (MPa ⁻¹) $\times 10^{-6}$	Uncertainty of measurement (MPa ⁻¹) $\times 10^{-6}$
LNE	-0.02	0.1	+0.04	0.11
IMGC	+0.81	0.3	-0.40	0.31
PTB	+1.01	0.3	-0.16	0.35
NPL	+3.2	0.2	-0.49	0.23
NBS	-0.72	0.3	-0.13	0.35
BEV	-6.02	1	+2.72	1.06
CSMU	+3.4	0.1	+0.14	0.16
FFA	-2.75	1.1	-0.71	1.12
EAM	+0.88	1	+0.23	1.01
NRLM	-0.558	0.03	-0.01	0.06
NIM	+2.9	0.15	-0.10	0.18
ASMW	+3.96	0.4	-0.05	0.45
VNIIFTRI	+2.76	0.06	-0.08	0.06

results were excluded, their value of k lying in the range 1.29 to 2.57.

Table 4 shows the pressure distortion coefficient of the laboratories' own standards; also given is the difference between each participant's measured value for the transfer standard relative to the reference value; these differences are also shown in Fig. 6. Overall, the differences lie within 3.43×10^{-6} MPa⁻¹, and eleven results lie within 0.72×10^{-6} MPa⁻¹.

5.7 Comparative Results at 100 MPa

Again using the acceptability test described above, the reference value was based on the results of ten laboratories, the value of k for those excluded lying in the range 1.01 to 2.42.

In practical terms, the difference in the measurement of A_p at 100 MPa represents the degree of dis-

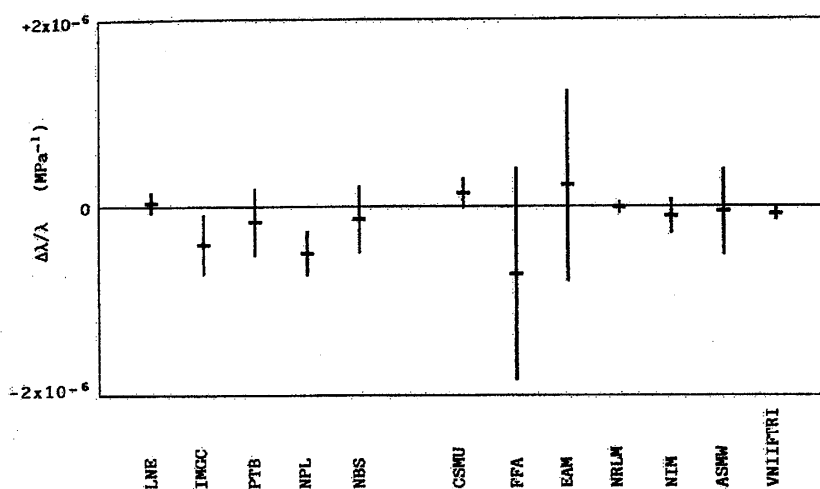


Fig. 6. Deviation of the participants' values of $\Delta\lambda$ from the reference values, expressed in MPa^{-1}

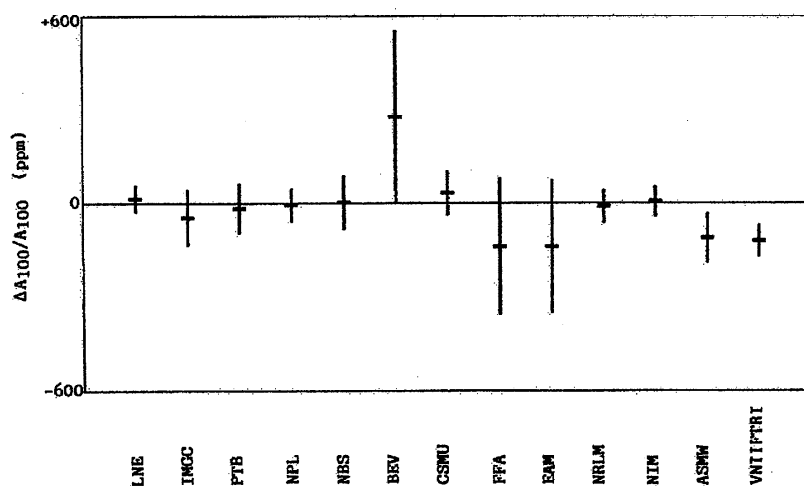


Fig. 7. Deviation of the participants' values of $\Delta A_{100}/A_{100}$ from the reference values, expressed in ppm

agreement between the participating laboratories when measuring pressure in the region of 100 MPa. Here the measurement of the pressure distortion coefficient plays a major role, and it can be seen from Table 5 (and Fig. 7) that the overall values lie within 414 ppm, whereas the results of eight of the laboratories lie within 78 ppm.

6. Conclusion

This comparison has highlighted the difficulties in measuring effective area, especially its dependence upon applied pressure. However, there is no evidence to indicate that there is any correlation between the design of the laboratory standard or the method of

Table 5. Measurement of the effective area of the transfer standard at 100 MPa

Laboratory	Difference of the reference value (expressed in ppm)	Uncertainty on the measurement of A_{110} (expressed in ppm)
LNE	+ 16.5	42
IMGC	- 45	88
PTB	- 15.3	79
NPL	- 4.6	52
NBS	+ 4.2	85
BEV	+277	276
CSMU	+ 33	70
FFA	-137	217
EAM	-137	212
NRLM	- 9.9	52
NIM	+ 6.4	47
ASMW	-110	78
VNIIFTRI	-119	49

deriving the pressure distortion coefficient with the result obtained.

The linearity (inside 5×10^{-6}) and the repeatability (a few ppm) encountered in this intercomparison allow the assumption that the accuracy of pressure balances will be increased at high pressure by a better knowledge of the pressure distortion coefficient.

The results have shown an overall agreement between participants of 400 ppm at 100 MPa, decreasing to about 200 ppm for lower pressures.

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